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Commissioner for Patents, P.O. Box 1450, Alexandria, VA. 22313-1450 on  
September 29, 2008

Dated: September 29, 2008

Signature: \_\_\_\_\_  
(Eileen Sheffield)

Docket No.: NY-CHEMMT-206.1-  
Cont.-US

(PATENT)

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of:  
Klaus-Dieter NITTEL, et al.

Application No.: 10/830,181

Group Art Unit: 1742

Filed: April 22, 2004

Examiner: Zheng, Lois L.

For: **METHOD FOR APPLYING MANGANESE  
PHOSPHATE LAYERS**

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA. 22313-1450

Dear Sir:

**DECLARATION UNDER RULE 132**

Sir:

1. I am Ralf Schneider, a materials' science investigator employed by the assignee of the present invention. I have three (3) years education in materials' science development and testing and attended five (5) years of evening school in mechanical engineering. I have been employed by Chemetall GmbH since 1995 and I have been working in the field of manganese phosphating since then.

2. I am familiar with the patent application, and I am familiar with the Office Action dated June 30, 2008.

3. I submit this declaration in view of the Examiner's analysis of the application, particularly the Examiner's 35 U.S.C. § 103(a) rejections of claims 8-10 and 13 over U.S. Patent No. 3,860,455 (Hansen) in view of U.S. Patent No. 2,375,468 (Clifford), and of the remaining

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claims over Hansen and Clifford in view of either U.S. Patent No. 5,795,408 (Bittner), U.S. Patent No. 4,824,490 (Oei) or U.S. Patent No. 2,987,427 (Shaw).

4. I have reviewed all of the cited references with a particular focus on Example 1 with the bath of Experiment C of Hansen et al. ("Hansen"), which relates to a manganese phosphating without nitroguanidine for the coating of steel.

5. The coating solutions used for the experiments according to Hansen, as described in the Declaration of October 1, 2007, but must be reworked as set forth below to obtain the necessary acid values. The data listed there for  $P_2O_5$  and for Ni content in Table 1 deviate from 36.9 to 36.7 g/L (Hansen) and from 0.34 to 0.33 g/L (Hansen), respectively. These data measured by Inductively Coupled Plasma (ICP) mass spectrometry Vista Pro of Varian with an optical spectral photometer. In the art of phosphating, it is typical to work and to rework any composition by adjusting primarily the acidity data. There, it was primarily tried to gain the data for Total Acid TA of 82 points. If such acidity data are not reached, usually either a small portion of the phosphating concentrate enriching the bath solution or a small portion of water diluting the bath solution are added to the coating solution. This also slightly influences the contents of cations and anions accordingly. Such handling of phosphating solutions is typical for this art. The differences between the measured data and the data of Hansen are quite small compared with measurement uncertainties allowed for such measurements and for such art. Furthermore, these small differences do not influence the properties of the solutions, of the process and of the properties of the coatings significantly as these vary only over broader chemical ranges.

6. As shown in my prior Declaration, the gasing time, which indicates the pickling time of the acidic solution on the metallic surface by the generation of hydrogen gas, is significantly reduced by the addition of nitroguanidine, which means that there is a significantly shorter coating time. Only during the pickling or substantially only during the pickling of a coating solution is hydrogen gas released, as are cations which are taken up from the coating solution. This means that from steel sheets primarily iron ions are dissolved. The cation content of a coating solution is to a large extent proportional to the sludge generation, especially for iron ions.

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7. If the pickling time of Hansen is typically about twice the time with coating solutions of Hansen in comparison to the coating solutions of the present invention, then the content of released iron cations is typically enriched by about twice as much in the coating solutions according to Hansen in comparison to those of the present invention. Because of the longer and therefore stronger pickling according to Hansen, the roughness  $R_z$  of the generated coatings is significantly higher.

8. A test to measure the sludge generation with a minimum of accuracy would require a throughput of at least  $40 \text{ m}^2$  of steel sheets for a bath of  $1 \text{ m}^3$  of volume. The bath volume of such an industrial phosphating is typically 1 to  $5 \text{ m}^3$ . Even in a laboratory, such experiments would require higher consumptions and efforts.

9. The teaching of the present application is specifically directed to gain a minimum content of  $\text{Fe}^{2+}$  and to limit its content by the addition of nitroguanidine. Nitroguanidine limits the pickling time, but does not work as an oxidant, e.g., for  $\text{Fe}^{2+}$  and does not remove small hydrogen bubbles from the metallic surface. Typical accelerators work as an oxidant, e.g., for  $\text{Fe}^{2+}$  and remove small hydrogen bubbles from the metallic surface. Therefore, nitroguanidine is no typical "accelerator". By the use of nitroguanidine in a certain concentration, there is a limitation of iron content in the coating solution and a limitation of the possibility to generate sludge. But Hansen does not give any hint or suggestion as to how to limit the iron content.

10. Concerning the coating results received from the reworking of the examples of Clifford, the SEM photographs have shown that there was generated no closed coating which is necessary for corrosion resistance or for coldforming and no visible coating was even generated, which would otherwise be well visualized under a Scanning Electron Microscope. An expert in the art would not work under the conditions of Clifford, as he knows that these conditions are not suitable.


11. In paragraph 11 of my previous declaration, 3 g/L of nitroguanidine were used for the experiments 3.) and 4.) according to Clifford, which correspond to about 0.3 % by weight of nitroguanidine. As the coating solution does not have a density of exactly 1.00 g/ml, the 0.3 % by weight do not correspond exactly to 3 g/L. This indicates that the nitroguanidine content used

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is nearly as described in Clifford. The starting bath 1.) had a free acidity FA of 13 points and a total acidity of 80 points. During the further experiments until 4.), these data did not vary much, but the exact data were not written down for the further experiments.

12. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

By 

Date 7-10-2008

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